

Preparation and Characterisation of Fe/Ce Multilayer

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Ce/Fe multilayer (ML) with constant Fe (2 nm) and Ce (4.5 nm) sublayer thicknesses was prepared onto naturally oxidised Si(100) substrate using magnetron sputtering. Chemical purity of the sublayers was revealed *in-situ* by X-ray photoelectron spectroscopy (XPS) measurements. The structure of the sample was studied by standard low- and high-angle X-ray diffraction (XRD). Surface morphology of the ML was examined by atomic force microscopy. Magnetic properties of the sample was studied in the temperature range between 5 and 350 K using a vibrating sample magnetometer in a magnetic field up to 9 T. The hysteresis loops were measured in field perpendicular and parallel to the substrate. Furthermore, hydrogen absorption at a pressure of about 1000 mbar was studied at room temperature (RT) in Pd covered ML using four-point resistivity measurements. The solid state amorphisation reaction have been confirmed by XRD and magnetic measurements of the Ce/Fe ML. The absence of satellite peaks in the low - angle XRD pattern revealed no artificial layered structure. The above results show that interdiffusion of cerium and iron atoms is extremely fast at RT.

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1. Introduction

It is well known that magnetic multilayers exhibit a rich variety of novel effects related to interface effects in ultrathin layers [1–3]. An important studies of magnetic layerd structures is the influence of a spacer layer inserted between two ferromagnetic layers. It has been shown that, depending on the thickness of the spacer layer, two ferromagnetic layers may be coupled parallel or antiparallel. Moreover, the induced magnetism of the spacer layer due to proximity effect has been also evidenced [4, 5].

In the layered Fe/Ce structures, for a thickness smaller than 2.5 nm, Ce adopts an α -like electronic configuration with a magnetic order [6] in contrast to the α - phase of Ce metal which is nonmagnetic. While the magnetic ordering is relatively well understood for elemental rare earths, the 5d magnetization of Ce is much more complex because of its 4f electronic states which are at the borderline between localization and itinerancy.

Light rare earth (RE) metals like La and Ce form a cubic dihydride REH₂ with hydrogen atoms on tetrahedrally coordinated sites (CaF₂ - type structure) and dissolve, in a single phase, further hydrogen on octahedral sites up to the cubic trihydride REH₃ (BiF₃ - type structure) [7]. The hydrides REH_x undergo a transition from a metal to an insulator as the hydrogen content approaches $x = 3$, with the accompanying change from high optical reflectivity to transparency for photon energies below 2 eV [7]. The transition is continuous and reversible, and a thin film of such material allows to rapidly switch be-

tween the contrasting optical properties of the dihydride and trihydride phase, simply by varying the surrounding hydrogen gas pressure or an electrolytic cell potential [8].

In this paper we study structure, morphology and interface alloying in Fe/Ce multilayer (ML). Moreover, hydrogen absorption kinetics at a pressure of about 1000 mbar was measured at room temperature (RT) in Pd covered multilayered sample.

2. Experimental procedure

Fe/Ce ML was prepared at RT using ultra high vacuum (UHV) (5×10^{-10} mbar) magnetron sputtering [9–11]. Schematic description of the prepared Fe/Ce ML is shown in Fig. 1. As a substrate we have used Si(100) wafers with naturally oxidised surface to prevent a silicide formation. Therefore we have applied a special heat treatment in UHV before deposition in order to obtain a smooth SiO_x surface layer. The Ce-layers ($d_{\text{Ce}} = 4.5$ nm) were deposited using a radio frequency (RF) current source. For preparation of the Fe-layers ($d_{\text{Fe}} = 2$ nm)

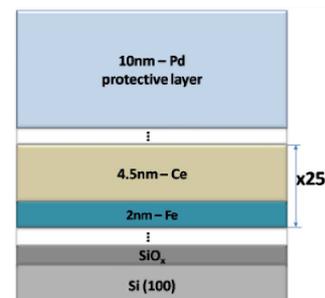


Fig. 1. Schematic description of the prepared Fe/Ce multilayer.

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a direct current (DC) source was used. The deposition rates of Fe and Ce were individually checked by quartz thickness monitors. The thickness of individual layers were controlled by varying their deposition times. Furthermore, the calibrations of the deposition rates were also determined by thickness measurements of reference samples using DEC-TAC (Bruker) profilometer and X-ray fluorescence analysis. The number of repetition was equal to 25. Finally, a protective layer of 10 nm Pd was deposited to allow a fast uptake and release of hydrogen at RT and to avoid oxidation of the top Ce sub-layer [12, 13].

The chemical composition of all the layers was checked *in-situ*, immediately after deposition, transferring the samples to an UHV (4×10^{-11} mbar) analysis chamber equipped with X-ray Photoelectron Spectroscopy (XPS). Details of the XPS measurements can be found in Ref. [14–16]. The XPS spectra were measured at RT using a SPECS EA 10 PLUS energy spectrometer with Al- K_{α} radiation of 1486.6 eV. The energy spectra of the electrons were analysed by a hemispherical analyser ($\text{FWHM}_{\text{Mg-K}\alpha} = 0.8$ eV for Ag- $3d_{5/2}$). All emission spectra were measured immediately after the sample transfer in vacuum of 8×10^{-11} mbar. The structure of the Fe/Ce ML was examined using standard $\theta - 2\theta$ X-ray diffraction (XRD). Hydrogen loading was performed in the HV chamber evacuated to about 10^{-6} mbar.

Absorption kinetics at a pressure of about 1000 mbar was studied at RT in Pd covered Fe/Ce ML using four-point resistivity measurements. The above procedure does not change the hysteresis loop of the “as prepared” sample.

The magnetic characterisation of the ML was carried out using a Vibrating Sample Magnetometer (VSM) in the temperature range $5 \div 350$ K in an in-plane and perpendicular magnetic field up to 9 T.

3. Results and discussion

XPS core-level spectra for the freshly prepared 2 nm Fe and 4.5 nm Ce thin films are shown in Fig. 2. As can be observed, the impurities are absent on the surfaces of such prepared thin films. Practically no XPS signal from potential contamination atoms like O-1s and C-1s was observed.

In Fig. 3 we show AFM image (300×300 [nm]) of Pd covered Fe/Ce ML measured at RT. The Pd grains show an in-plane texture because the magnetron sources are mounted at an angle of about 60 deg to the substrate. However, the surface of the ML is very smooth. We have estimated an average roughness parameter R_a from a greater area (2000×2000 [nm]) as small as 0.22 nm.

Figure 4 shows low- and high-angle XRD patterns of the Fe/Ce ML. The positions of the observed reflections (Fig. 4a) reveal formation of an amorphous alloy due to solid state reaction already during the sample growth process at RT. The above result is in good agreement with earlier studies reported in Ref. [17]. In the high-angle diffraction pattern (Fig. 4b) we have observed the

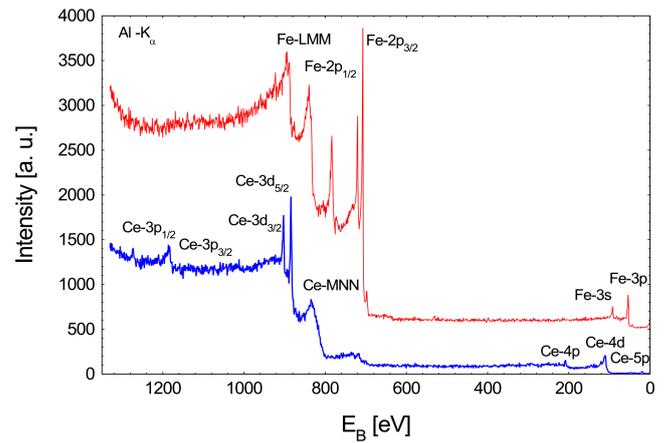


Fig. 2. XPS (Al- K_{α}) spectra of *in-situ* prepared 2 nm Fe and 4.5 nm Ce thin films.

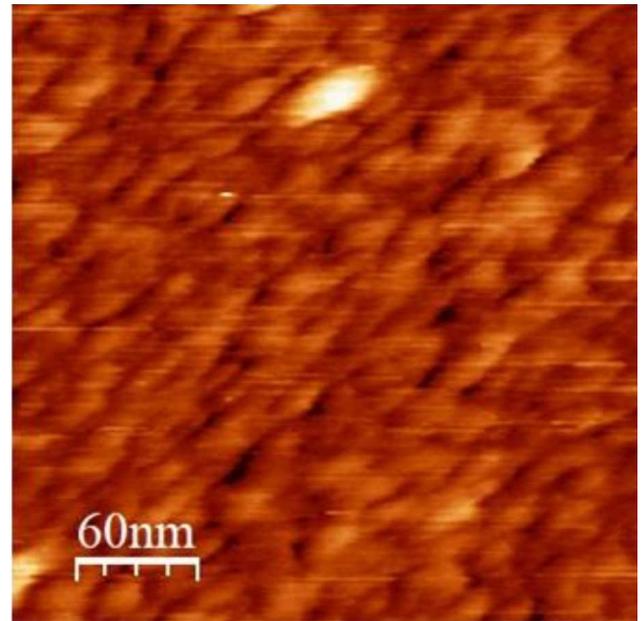


Fig. 3. AFM image of (2nm Fe/4.5nm Ce) \times 25/10 nm Pd ML. Average roughness parameter R_a was estimated as about 0.22 nm.

only one peak near 40 deg originated from textured Pd protective layer. Other XRD signal between 30 and 35 deg originates from oxidised Si substrate as shown in Fig 4b. We also haven’t observed typical for crystalline MLs central Bragg peak because formation of an amorphous Fe-Ce alloy due to solid state reaction [18].

The hysteresis loops measured for Fe/Ce ML in an in-plane and perpendicular to the substrate magnetic field are shown in Fig. 5a and Fig. 5b, respectively. The measurements were performed at 5 K (solid line) and 300K (broken line). The relatively high drop of the saturation magnetisation at 300 K and low saturation field measured in direction perpendicular to the substrate also revealed formation of an amorphous Fe-Ce alloys during the sample deposition.

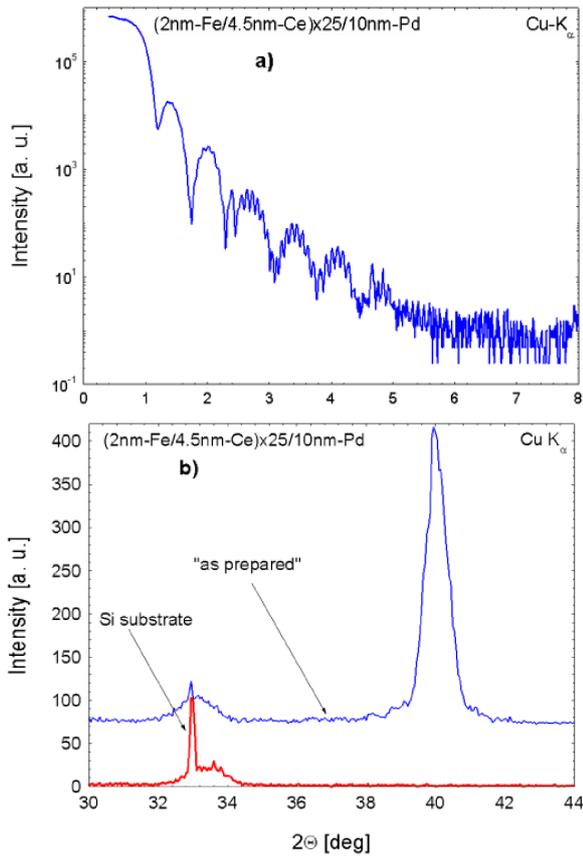


Fig. 4. Low- (a) and high-angle (b) XRD (Cu-K α) pattern for (2nm Fe/4.5 nm-Ce) \times 25/10 nm Pd multilayer. For a comparison XRD pattern for Si(100) substrate with naturally oxidised surface is also shown in part (b).

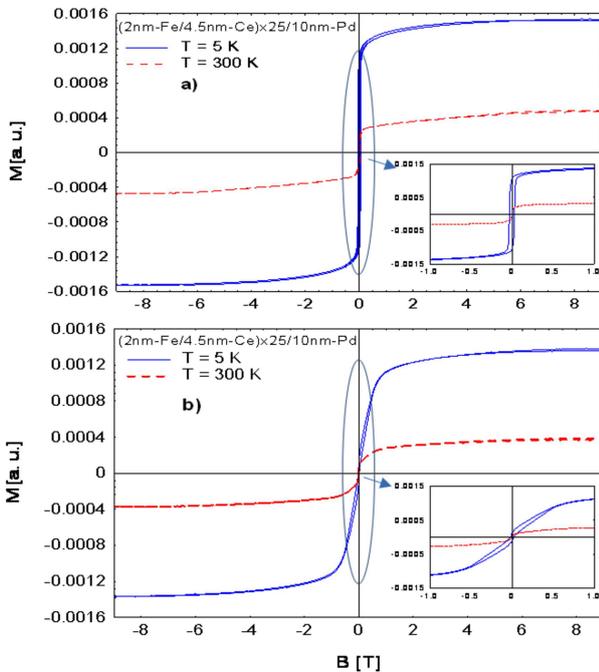


Fig. 5. Hysteresis loops measured parallel (a) and perpendicular (b) to the plane for (2 nm Fe/4.5 nm Ce) \times 25/10 nm Pd multilayer.

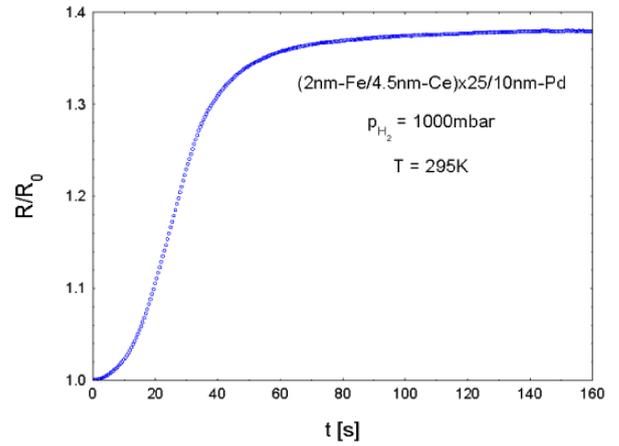


Fig. 6. Relative resistance (R/R_0) as a function of hydrogenation time at a pressure of 1000 mbar measured at room temperature for (2 nm Fe/4.5 nm Ce) \times 25 multilayer covered by 10 nm of Pd protective layer. $R_0 = R(t = 0)$.

In Fig. 6 we show relative resistance (R/R_0) of the Fe/Ce ML as a function of hydrogenation time at a pressure of about 1000 mbar measured at RT. Results showed that the fastest initial rise in resistance was observed in the first \sim 40 s of the hydrogenation.

In conclusion, the solid state amorphisation reaction was confirmed by XRD and magnetic measurement in the “as-prepared” Ce/Fe ML. The above results show that interdiffusion of cerium and iron is extremely fast at RT. Furthermore, it was observed a relatively fast resistivity change of the ML during the first 40 s of hydrogen absorption at room temperature.

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